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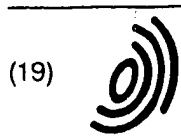
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(54) **STEEL MATERIAL HAVING HIGH DUCTILITY AND HIGH STRENGTH AND PROCESS FOR PRODUCTION THEREOF**

(57) A steel product having a structure composed mainly of ferrite or ferrite plus pearlite or ferrite plus cementite. A steel pipe produced from this steel product by rolling at a ferrite recrystallization temperature such that the reduction of area is greater than 20%. This steel pipe is characterized by grain size not greater than 3  $\mu$ m, preferably not greater than 1  $\mu$ m, elongation greater than 20%, tensile strength (TS : MPa) and elongation (EI : %) whose product is greater than 10000, and percent ductile fracture greater than 95%, preferably 100%, measured by Charpy impact test on an actual pipe at -100°C. The structure is characterized by C : 0.005-0.03%, Si : 0.01-3.0%, Mn : 0.01-2.0%, and Al : 0.001-0.10% on a weight basis, and is composed of ferrite or ferrite and a secondary phase, with ferrite grains being not greater than 3  $\mu$ m and the secondary phase having an areal ratio not more than 30%. The steel pipe is produced from a steel pipe stock having the above-mentioned composition by heating at a temperature of (Ac<sub>1</sub>  $\pm$  50°C) to 400°C and subsequently performing reduc-

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## Description

### Technical Field

5 [0001] The present invention relates to a steel product which has high strength and high ductility and is superior in toughness and resistance to collision and impact, particularly a steel product, such as steel pipe, wire rod, steel bar, steel section, steel plate, and steel strip, having fine crystal grains, and also to a process for production thereof.

### Background Art

10 [0002] Common practice to increase the strength of a steel product is to add an alloying element such as Mn and Si, to perform heat treatment such as controlled rolling, controlled cooling, quenching, and tempering, and to add a precipitation hardening element such as Nb and V. However, what is required of steel products is not only high strength but also high ductility and high toughness. There has been a demand for a steel product which has well-balanced strength  
15 and ductility/toughness.

[0003] Making grains finer is one of a few important means to improve both strength and ductility/toughness. This is accomplished by performing austenite-ferrite transformation from fine austenite while preventing austenite grains from becoming coarse, thereby giving fine ferrite grains, by working which makes austenite grains finer, thereby giving fine ferrite grains, or by utilizing martensite and lower bainite that result from quenching and tempering.

20 [0004] One of these methods in general use for steel production is controlled rolling which consists of strengthening in the austenite region and its ensuing austenite-ferrite transformation to give rise to fine ferrite grains. Another way in practice is to add a trace amount of Nb which suppresses the recrystallization of austenite grains, thereby yielding finer ferrite grains. Working at a temperature at which austenite does not yet recrystallize permits austenite grains to grow, giving rise to the deformation zone in grains, and finer ferrite grains occur from this deformation zone. A recent practice  
25 to obtain finer ferrite grains is controlled cooling that is carried out during or after working.

[0005] The above-mentioned methods, however, need rebuilding of the existing facilities and considerable remodeling of the current process in the production of steel products, such as steel pipes, having improved collision and impact resistance required for better automotive safety, an ever increasing demand. Therefore, they are economically unfeasible.

30 [0006] In the meantime, steel products for line pipe need resistance to stress corrosion cracking by sulfides, and this object is achieved by hardness control through the reduction of impurities or the adjustment of alloying elements. In addition, conventional practices to improve fatigue resistance include heat treatment, such as thermal refining, induction hardening, and carburizing, and addition of a large amount of expensive alloying elements such as Ni, Cr, and Mo. The disadvantage of these methods is poor weldability and high production cost.

35 [0007] Steel pipes of small to medium diameter are produced mainly by electric resistance welding with high frequency current. The process for their production consists of continuously feeding a flat strip steel, making it into a pipe stock using a forming roll, heating the opposing edges of the pipe stock to a temperature above the melting point of steel by means of high frequency current, and butt-welding the heated edges by means of squeeze rolls.

[0008] This process, however, has a disadvantage of requiring rolls that conform to the dimensions of the desired  
40 steel pipe; therefore, it is not suitable for multi-product production in small lots.

[0009] In order to address this problem, there has been proposed a new process in, for example, Japanese Patent Publication No. 24606/1990. This process consists of heating a flat strip steel in a preheating furnace and a heating furnace, making the strip steel into a pipe by electric resistance welding, heating the pipe to a temperature above the  $A_3$  transformation point, and rolling the heated pipe by a reducing mill so that it has a predetermined outside diameter.

45 [0010] This process, however, poses problems due to heating above  $A_3$  point. Heating gives rise to scale which is bitten during rolling. Heating also makes crystal grains coarse, aggravating the ductility, strength, and toughness of the resulting steel pipe.

[0011] A cold sizing process has been proposed in Japanese Patent Laid-open No. 33105/1988. This process is designed to reduce the outside diameter of a hollow pipe stock, such as seamless steel pipes and electric welded pipes,  
50 in the cold state by using a series of reducing mills, each consisting of three rolls. The disadvantage of this process is the necessity of a large-scale mill to withstand high loads due to cold rolling and the necessity of a lubricating facility to prevent rolls from seizing. In addition, cold rolling gives rise to working strain, which aggravates ductility and toughness.

[0012] It is an object of the present invention, which was completed to address the above-mentioned problems, to provide a steel product and a process for production thereof, said steel product being superior in ductility, strength,  
55 toughness, and resistance to collision and impact owing to fine ferrite crystal grains.

one or more selected from REM not more than 0.02% and Ca not more than 0.01%, with the remainder being Fe and unavoidable impurities.

[0024] The above-mentioned composition may be C 0.005-0.30%, Si 0.01-3.0%, Mn 0.01-2.0%, Al 0.001-0.10%, and one or more selected from Cu not more than 1%, Ni not more than 2%, Cr not more than 2%, and Mo not more than 1% and one or more selected from Nb not more than 0.1%, V not more than 0.3%, Ti not more than 0.2%, and B not more than 0.004%.

[0025] The above-mentioned composition may be C 0.005-0.30%, Si 0.01-3.0%, Mn 0.01-2.0%, Al 0.001-0.10%, and one or more selected from Cu not more than 1%, Ni not more than 2%, Cr not more than 2%, and Mo not more than 1%, and one or more selected from REM not more than 0.02% and Ca not more than 0.01%, with the remainder being Fe and unavoidable impurities.

[0026] The above-mentioned composition may be C 0.005-0.30%, Si 0.01-3.0%, Mn 0.01-2.0%, Al 0.001-0.10%, and one or more selected from Nb not more than 0.1%, V not more than 0.3%, Ti not more than 0.2%, and B not more than 0.004%, and one or more selected from REM not more than 0.02% and Ca not more than 0.01%, with the remainder being Fe and unavoidable impurities.

[0027] Moreover, the above-mentioned composition may be C 0.005-0.30%, Si 0.01-3.0%, Mn 0.01-2.0%, Al 0.001-0.10%, one or more selected from Cu not more than 1%, Ni not more than 2%, Cr not more than 2%, and Mo not more than 1%, one or more selected from Nb not more than 0.1%, V not more than 0.3%, Ti not more than 0.2%, and B not more than 0.004%; and one or more selected from REM not more than 0.02% and Ca not more than 0.01%, with the remainder being Fe and unavoidable impurities.

[0028] The present invention also covers a process for producing a steel pipe with high ductility and high strength, said process comprising heating a pipe stock having any of the above-mentioned compositions at ( $Ac_1 + 50^\circ C$ ) to  $400^\circ C$ , preferably  $750-400^\circ C$ , and reducing the heated pipe stock at ( $Ac_1 + 50^\circ C$ ) to  $400^\circ C$ , preferably  $750-400^\circ C$ , such that the cumulative diameter reduction is 20% or more. The rolling is preferably carried out such that at least one pass reduces the diameter by 6% or more per pass and the cumulative diameter reduction is 60% or more. In addition, the reducing mentioned above is preferably carried out by the aid of lubrication.

[0029] The present inventors also found that the above-mentioned process permits the production of a steel pipe with high strength and high toughness and superior resistance to stress corrosion cracking if the composition of the pipe stock is specified in an adequate range. This finding led the present inventors to conceive to utilize the process for the production of line pipes.

[0030] Line pipes conventionally have the content of impurities, such as S, reduced and the hardness controlled by means of alloying elements for improvement in resistance to stress corrosion cracking. Such conventional methods are limited in strengthening and pose a problem with high production cost. Specifying the composition of the pipe stock in an adequate range and performing the reduction in the ferrite recrystallizing region yield a line pipe with high strength and high toughness, owing to dispersed fine ferrite and fine carbide, superior in resistance to stress corrosion cracking resistance due to limited alloying elements, leading to reduced hardening by welding and less crack generation and propagation.

[0031] Accordingly, the present invention covers a process for producing a steel pipe superior in ductility and resistance to collision and impact as well as resistance to stress corrosion cracking resistance, said process comprising heating a pipe stock at ( $Ac_1 + 50^\circ C$ ) to  $400^\circ C$ , preferably  $750-400^\circ C$ , and reducing the heated pipe stock at ( $Ac_1 + 50^\circ C$ ) to  $400^\circ C$ , preferably  $750-400^\circ C$ , such that the cumulative diameter reduction is 20% or more, said pipe stock having a composition of C 0.005-0.10%, Si 0.01-0.5%, Mn 0.01-1.8%, Al 0.001-0.10%, one or more selected from Cu not more than 0.5%, Ni not more than 0.6%, Cr not more than 0.5%, and Mo not more than 0.5%, and one or more selected from Nb not more than 0.1%, V not more than 0.1%, Ti not more than 0.1%, and B not more than 0.004%, or further one or more selected from REM not more than 0.02% and Ca not more than 0.01%, with the remainder being Fe and unavoidable impurities.

[0032] The present inventors also found that the above-mentioned process permits the production of a steel pipe with high strength and high toughness and superior fatigue resistance if the composition of the pipe stock is specified in an adequate range. This finding led the present inventors to conceive to utilize the process for the production of steel pipes with high fatigue resistance. Specifying the composition of the pipe stock in an adequate range and performing the reduction in the ferrite recrystallizing region yield a steel pipe with high strength and high toughness, owing to dispersed fine ferrite and fine precipitation, superior in fatigue resistance due to limited alloying elements, leading to reduced hardening by welding and less crack generation and propagation.

[0033] Accordingly, the present invention covers a process for producing a steel pipe superior in ductility and strength as well as fatigue resistance, said process comprising heating a pipe stock at ( $Ac_1 + 50^\circ C$ ) to  $400^\circ C$ , preferably  $750-400^\circ C$ , and reducing the heated pipe stock at ( $Ac_1 + 50^\circ C$ ) to  $400^\circ C$ , preferably  $750-400^\circ C$ , such that the cumulative diameter reduction is 20% or more, said pipe stock having a composition of C 0.06-0.30%, Si 0.01-1.5%, Mn 0.01-2.0%, and Al 0.001-0.10%, with the remainder being Fe and unavoidable impurities.

that the austenitic change is 25% or less.

[0039] The rolling temperature is restricted to a range in which ferrite recrystallization takes place. In the present invention, this range is preferably 400-750°C, depending on the chemical composition of the steel blank used. Rolling at a temperature higher than this range gives rise to a two-phase structure of ferrite plus austenite containing a large amount of austenite or a single-phase structure of austenite. The resulting product does not have the structure composed mainly of ferrite or ferrite plus pearlite or ferrite plus cementite. On the other hand, rolling at a temperature exceeding 750°C causes ferrite grains to grow remarkably after recrystallization. This is detrimental to the desired fine grains not greater than 3 µm, preferably not greater than 2 µm. Rolling at a temperature lower than 400°C is difficult to carry out due to blue shortness, with decrease in ductility and toughness on account of insufficient recrystallization and residual deformation strain. Therefore, the rolling temperature is 400-750°C, preferably 560-720°C, more preferably 600-700°C. At 560-720°C, the grain size will be not greater than 1 µm, and at 600-700°C, the grain size will be not greater than 0.8 µm. Fig. 3 schematically shows the relation between the grain size and the rolling temperature (at the start and end of rolling).

[0040] Rolling is carried out such that the reduction of area is greater than 20%. In the present invention, the reduction of area is defined as the value calculated by the formula  $(A_0 - A)/A \times 100$ , where  $A_0$  is the cross sectional area before rolling and A is the cross sectional area after rolling. With a reduction of area less than 20%, rolling does not make recrystallized grains finer because of insufficient strain. The reduction of area is preferably greater than 50%.

[0041] After rolling, the steel product is cooled to room temperature. Cooling may be natural air cooling or any of known forced air cooling, water cooling, and mist cooling. The latter is desirable to suppress the growth of grains. The cooling rate is preferably greater than 1°C/s.

[0042] An appropriate rolling method may be selected according to the shape of the stock. For steel pipe stocks, reducing by means of a plurality of grooved rolls, called as a reducer, is desirable. Stocks adequate for this process include electric resistance welded pipes, forge-welded steel pipes, and solid phase pressure-welded steel pipes.

[0043] According to the present invention, rolling is carried out with lubrication. Lubricated rolling ensures uniform distribution of strain and grain size in the thickness direction. Rolling without lubrication tends to cause concentrated strain in the surface and uneven grain size distribution in the thickness direction. Ordinary rolling oils, such as mineral oil and synthetic ester, may be used for lubricated rolling. They are not specifically restricted.

[0044] The above-mentioned process yields a high-toughness, high-ductility steel product which has a structure composed mainly of ferrite or ferrite plus pearlite or ferrite plus cementite, and which has an average grain size not greater than 3 µm, preferably not greater than 1 µm, in the cross section perpendicular to the lengthwise direction of the steel product. The steel product of the present invention may have a structure which contains not more than 30% of bainite in addition to ferrite, pearlite, and cementite. The steel product will increase in strength but decrease in toughness and ductility if it contains bainite more than specified above and martensite.

[0045] With an average grain size in excess of 3 µm, the steel product will lose a balance between strength and toughness/ductility; that is, it does not meet the requirement that elongation is 20% or more and the product of tensile strength (TS: MPa) and elongation (El: %) is 10000 or more. A large average grain size leads to brittle cracking that occurs in the cross section in the lengthwise direction of the steel pipe during Charpy impact test at -100°C. This implies a failure to meet the requirement for toughness that the percent ductile fracture is 95% or more, preferably 100%. With an average grain size not greater than 3 µm, preferably not greater than 1 µm, the steel pipe is less vulnerable to brittle cracking in the cross section perpendicular to the lengthwise direction and is superior in toughness.

[0046] The process of the present invention for producing steel products will be described in more detail in the following, with stress placed on steel pipes.

[0047] The present invention employs steel pipes as the stock. There are no specific restrictions on the process of producing steel pipe stocks. Adequate examples include electric resistance welded steel pipes produced by electric resistance with high frequency current, solid-phase pressure-welded steel pipes produced by pressure welding after heating edges to a temperature suitable for solid-phase pressure-welding, forge-welded steel pipes, and seamless steel pipes produced by Mannesmann piercing rolling.

[0048] The following explains the reason why the chemical composition is restricted for the steel pipes as stock and product.

C : 0.005-0.30%

[0049] C is an element which dissolves in the basic metal to form a solid solution or precipitates in the form of carbide in the basic metal, thereby increasing the strength of steel. Cementite, martensite, and bainite that precipitate in the form of fine grains as the hard secondary phase contribute to ductility (uniform elongation). For the desired strength and ductility due to cementite that precipitates as the secondary phase, the content of C is 0.005% or more, preferably 0.04% or more. C in excess of 0.30% increases strength so much as to adversely affect ductility. Therefore, the content of C is limited to 0.005-0.30%, preferably 0.04-0.30%. Moreover, the content of C is not more than 0.10% for the

the content of B is not more than 0.004%, preferably 0.0005-0.002%.

[0061] Incidentally, each content of Ni, V, and Ti is not more than 0.1% for line pipes to have improved resistance to stress corrosion cracking. When used in excess of 0.1%, they adversely affecting resistance to stress corrosion cracking due to precipitation hardening.

REM : not more than 0.02% and Ca : not more than 0.01%.

[0062] Both REM and Ca adjust the form of inclusions and improve workability. They also precipitate in the form of sulfide, oxide or oxysulfide, thereby preventing the joints of steel pipe from becoming hard. They may be used alone or in combination with one another. When used excessively, they give rise to excessive inclusions, which lower cleanliness and adversely affect ductility. The content of REM is 0.004-0.02% and the content of Ca is 0.001-0.01%.

[0063] The above-mentioned composition for the steel pipe stock and steel product may additionally contain Fe as a remainder and unavoidable impurities as follows.

[0064] Unavoidable impurities are N : not more than 0.010%, O : not more than 0.006%, P : not more than 0.025%, and S : not more than 0.020%.

N : not more than 0.010%

[0065] N in an amount up to 0.010% is permissible, which is enough to form fine grains in combination with Al; however, excessive N adversely affects ductility. The content of N is not more than 0.010%, preferably 0.002-0.006%.

O : not more than 0.006%

[0066] O in an amount up to 0.006% is permissible. The content of O is as low as possible, because O forms oxides which adversely affect cleanliness.

P : not more than 0.025%

[0067] P segregates at grain boundaries, thereby adversely affecting toughness. The content of P is as low as possible, although up to 0.025% is permissible.

S : not more than 0.020%

[0068] S in an amount up to 0.020% is permissible. The content of S is as low as possible, because S forms sulfides which adversely affect cleanliness.

[0069] The following concerns the structure of the steel pipe as the product.

[0070] The steel pipe of the present invention is characterized by its structure composed of ferrite grains not larger than 3  $\mu\text{m}$ , preferably not larger than 1  $\mu\text{m}$ , so that it is superior in ductility and collision and impact resistance. With ferrite grains coarser than 3  $\mu\text{m}$ , the steel pipe will not have remarkably improved ductility and collision and impact resistance. The ferrite grain size is expressed in terms of average value of 200 or more ferrite grains regarded as circles which are observed under an optical or electron microscope when the cross section perpendicular to the lengthwise direction of the steel pipe is corroded with nitral solution.

[0071] In the present invention, the structure composed mainly of ferrite includes the one which is composed of ferrite alone without secondary phase and the one which is composed of ferrite and a secondary phase other than ferrite. The secondary phase other than ferrite includes martensite, bainite, and cementite. They may precipitate alone or in combination with one another. The secondary phase should have a ratio of area not more than 30%. The secondary phase that has precipitated helps elongation to occur evenly at the time of deformation, thereby improving the ductility and collision and impact resistance of the steel pipe. This effect becomes less significant as its ratio of area exceeds 30%. Fig. 4 shows an example of the structure of the steel pipe of the present invention.

[0072] The following concerns the process for producing the steel pipe of the present invention.

[0073] The process starts with heating the steel pipe stock having the above-mentioned composition. The heating temperature is ( $A_{c1} + 50^\circ\text{C}$ ) to  $400^\circ\text{C}$ , preferably  $750-400^\circ\text{C}$ . Heating beyond the upper limit deteriorates the surface properties and unduly increases austenite, resulting is coarse grains. Therefore, the heating temperature is not higher than ( $A_{c1} + 50^\circ\text{C}$ ), preferably not higher than  $750^\circ\text{C}$ . Heating below the lower limit does not provide an adequate rolling temperature. Therefore, the heating temperature is preferably  $400^\circ\text{C}$  or higher.

[0074] The heated steel pipe stock subsequently undergoes reducing preferably by a reducing mill of 3-roll type or 4-roll type or any other types. Continuous reducing by a plurality of stands is preferable. The number of stands depends on the dimensions of the steel pipe stock and finished steel pipe.

well-balanced strength and toughness/ductility. Sample No. 1-2, which underwent lubricated rolling, shows only a little variation in grain size in the thickness direction. In contrast, sample Nos. 1-4 and 1-5 (in comparative example) are poor in ductility and toughness due to coarse grains. Incidentally, it was found that pearlite (P) includes, in addition to the lamellar structure, pseudo pearlite which does not form the lamellar structure.

### Example 2

[0084] A steel raw material having the chemical composition shown in Table 1 was made into flat strip steel of 3.2 mm in thickness by hot rolling. This strip steel was continuously formed into an open pipe by means of a plurality of forming rolls. The open pipe had its edges preheated above the melting point by induction heating, and the edges were butt-welded by using squeeze rolls. Thus there was obtained a pipe stock, 31.8 mm in diameter and 3.2 mm in wall thickness. With its bead removed by a bead cutter, the resulting electric welded pipe was heated again at the temperature shown in Table 3 by induction heating. It was reduced by means of a 3-roll reducing mill to form a finished pipe having the outside diameter shown in Table 3.

[0085] The finished pipe thus obtained was tested for characteristic properties, i.e., structure, grain size, tensile properties, and toughness, in the same manner as in Example 1. The results are shown in Table 3.

[0086] It is noted from Table 3 that samples (Nos. 2-2, 2-3, 2-5, and 2-7) in examples pertaining to the present invention are characterized by fine grains not greater than 3  $\mu\text{m}$  and also by high elongation and toughness and well-balanced strength and toughness/ductility. By contrast, samples (Nos. 2-1, 2-4, 2-6, 2-8, and 2-9) in comparative examples are poor in ductility and toughness due to coarse grains.

### Example 3

[0087] A steel having the composition shown in Table 1 was prepared by using a converter, and this steel was made into a billet by the continuous casting process. After heating, this billet was made into a seamless pipe of 158 mm in outside diameter and 8 mm in wall thickness by using a Mannesmann mandrel mill. This seamless pipe was heated again to the temperature shown in Table 4 by induction heating and then reduced by means of a 3-roll reducing mill to form a product pipe having the outside diameter shown in Table 4.

[0088] The product pipe thus obtained was tested for characteristic properties in the same manner as in Examples 1 and 2. The results are shown in Table 4.

[0089] It is noted from Table 4 that samples (Nos. 3-1, 3-2, 3-4, and 3-5) in examples pertaining to the present invention are characterized by fine grains not greater than 3  $\mu\text{m}$  and also by high elongation and toughness and well-balanced strength and toughness/ductility. By contrast, samples (Nos. 3-3 and 3-6) in comparative examples are poor in ductility and toughness due to coarse grains.

### Example 4

[0090] A base steel pipe having the chemical composition shown in Table 5 was heated by induction to a temperature shown in Table 6 and then rolled into a finished steel pipe by means of a 3-roll reducing mill under the rolling conditions shown in Table 6.

[0091] The base steel pipe in Table 6 is either solid-phase pressure-welded one or seamless one. The former was prepared by preheating a 2.6 mm thick hot-rolled strip steel to 600°C, continuously forming it into an open pipe by means of a plurality of forming rolls, preheating the edges of the open pipe to 1000°C by induction, heating the edges to 1450°C below the melting point by induction, and pressure-welding the edges by means of a squeeze roll. It is 42.7 mm in diameter and 2.6 mm in wall thickness. The seamless pipe was prepared by using a Mannesmann mandrel mill from a continuously cast billet (with heating).

[0092] The product pipe thus obtained was tested for tensile properties, collision and impact properties, and structure. The results are shown in Table 6. Tensile properties were measured by using JIS No.11 specimens. Incidentally, elongation (EI) is expressed in terms of values calculated from

$$EI = EI_0 \times (\sqrt{a_0/a})^{0.4}$$

(where  $EI_0$  is the actually measured elongation,  $a_0$  is 292 mm<sup>2</sup>, and  $a$  is the sectional area (mm<sup>2</sup>) of the specimen.) This converted value was used in consideration of the size effect of the specimen. Collision and impact properties were evaluated in terms of the amount of energy which is absorbed before the amount of strain reaches 30% in the stress-strain curve obtained by the high-speed tensile test at a strain rate of 2000 s<sup>-1</sup>. Incidentally, collision and impact properties are a measure of energy required to deform the material when an automobile actually collides at a strain rate of 1000-2000 s<sup>-1</sup>. The larger the amount of this energy, the better the collision and impact resistance.

poor in energy absorption at the time of collision and impact, and poor in sulfide stress corrosion cracking resistance.

#### Example 7

[0105] A base steel pipe having the chemical composition shown in Table 11 was heated by induction to a temperature shown in Table 12 and then rolled into a product steel pipe by means of a 3-roll reducing mill under the rolling conditions shown in Table 12. The base steel pipe in this example was either electric resistance welded pipe of 110 mm in diameter and 2.0 mm in wall thickness or seamless steel pipe of 110 mm in diameter and 3.0 mm in wall thickness. The former was prepared by forming an open pipe from hot-rolled strip steel by means of a plurality of forming rolls and then welding the edges by induction heating. The latter was prepared by using a Mannesmann mandrel mill from a continuously cast billet with heating.

[0106] The product pipe thus obtained was tested for tensile properties, collision and impact properties, structure, and fatigue resistance. The results are shown in Table 12. Tensile properties and collision and impact properties were measured in the same manner as in Example 4. Fatigue strength was measured by subjecting the finished pipe as a specimen to cantilever reversed fatigue test (at a repeating rate of 20 Hz) in the air.

[0107] It is noted from Table 12 that samples (Nos. 7-1, 7-3, and 7-6 to 7-8) in examples have well-balanced ductility and strength, high tensile strength at high strain rate, and high energy absorption at the time of collision and impact. In addition, they are superior in fatigue resistance. By contrast, samples (Nos. 7-2, 7-4, and 7-5) in comparative examples are poor in fatigue strength. Sample No. 7-2 did not undergo reducing, sample 7-5 had a ratio of reduction in diameter which is outside the specified range, and sample No. 7-4 was reduced at a temperature outside the specified range. Therefore, it is poor in balance between strength and ductility due to coarse ferrite grains, poor in energy absorption at the time of collision and impact, and poor in fatigue resistance.

#### Exploitation in Industry

[0108] The present invention provides a high-strength steel product superior in toughness and ductility on account of extremely fine grain size not greater than 3  $\mu\text{m}$ . Therefore, it will produce a significant industrial effect of expanding the application area of steel products. The present invention also provides a process for efficient and easy production of high-strength steel pipe superior in ductility and impact resistance. Therefore, it will produce a significant industrial effect of expanding the application area of steel pipe. The present invention permits the production of steel pipes for line pipes which need high strength and toughness and good stress corrosion cracking resistance. The present invention also permits the economical production of high-strength, high-ductility steel pipe having good fatigue resistance, with the amount of alloying elements reduced.

Table 1

Steel	Chemical composition (wt%)						
No.	C	Si	Mn	P	S	Al	N
A	0.06	0.05	0.35	0.018	0.019	0.028	0.0025
B	0.08	0.25	1.28	0.007	0.002	0.041	0.0025
C	0.25	0.20	0.82	0.012	0.007	0.010	0.0028
D	0.16	0.22	0.75	0.009	0.006	0.031	0.0033

Table 3

Sample No.	Steel No.	O.D. of base pipe (mm)	Rolling conditions				O.D. of product pipe (mm)	Characteristics of product pipe							Note
			Heating temp. (°C)	Temp. at starting rolling (°C)	Temp. at finishing rolling (°C)	Reduction of area (%)		Grain size (μm)	Yield point YS (MPa)	Tensile strength TS (MPa)	Elongation El. (%)	TS x El (MPa·%)	Percent ductile fracture by Charpy (%)	Structure *	
2-1	B	31.8	900	770	715	60	15	20	430	457	29	13253	30	F+P+5%B	C. Ex.
2-2			800	770	715	60		2	470	500	40	20000	100	F+C+15%B	Ex.
2-3			700	670	630	60		1	523	556	39	21684	100	F+P+10%B	Ex.
2-4			700	520	535	60		6	619	658	13	8554	10	F+P+10%B	C. Ex.
2-5			600	590	605	60		1	581	618	36	22248	100	F+P+5%B	Ex.
2-6			520	500	520	60		6	620	660	14	9240	20	F+P+5%B	C. Ex.
2-7			700	660	630	30		3	502	534	33	17622	100	F+P+10%B	Ex.
2-8			700	660	650	10		9	435	468	30	14040	50	F+P+10%B	C. Ex.
2-9			-					31.8	12	424	460	29	13340	40	F+P+5%B

\* F: ferrite, P: pearlite (including pseudo pearlite), C: cementite, B: Bainite  
 Ex.: Example pertaining to the present invention. C. Ex.: Comparative Example

Table 5

Steel No.	Chemical composition (wt%)								Ac <sub>1</sub> (°C)	Note
	C	Si	Mn	P	S	Al	N	O		
E	0.09	0.40	0.80	0.012	0.005	0.035	0.0035	0.0025	770	Example
F	0.08	0.07	1.42	0.015	0.011	0.036	0.0038	0.0036	760	Example
G	0.06	0.21	0.35	0.013	0.008	0.028	0.0025	0.0028	775	Example
H	0.11	0.22	0.45	0.017	0.013	0.018	0.0071	0.0035	775	Example
I	0.21	0.20	0.50	0.016	0.013	0.024	0.0043	0.0030	770	Example
J	0.03	0.05	0.15	0.021	0.007	0.041	0.0026	0.0038	780	Example
K	0.09	0.15	0.52	0.024	0.003	0.004	0.0025	0.0026	775	Example

Table 6 (2)

No.	Steel No.	Base pipe		Rolling conditions								O.D. of product pipe (mm)	Characteristics of product pipe						Note
		Type	O.D. (mm)	Heating temp. (°C)	Temp. at starting rolling (°C)	Temp. at finishing rolling (°C)	Cumulative diameter reduction (%)	No. of total passes	No. of passes (5% and up per pass)	Final rolling speed (mm/min)	Tensile strength TS (MPa)		Elongation B (%)	High speed tensile strength (MPa)	Energy absorbed at collision and impact (MJ·m <sup>-2</sup> )	Ferrite grain size (μm)	Ratio of area of secondary phase (%)	Kind of secondary phase	
4-11	F	ERW	42.7	650	650	622	65	14	9	200	555	42	792	265	1.0	15	C	Ex.	
4-12	F	ERW	42.7	600	590	580	65	14	9	200	611	37	850	289	1.0	15	C	Ex.	
4-13	G	ERW	42.7	650	640	620	65	14	9	200	492	42	685	225	2.5	7	C	Ex.	
4-14	H	SP	110	700	695	670	77	17	10	150	475	52	666	219	2.0	9	C	Ex.	
4-15	I	SP	110	700	695	670	77	17	10	150	526	46	733	231	2.0	22	C+B	Ex.	
4-16	E	SP-PW	42.7	550	540	528	85	14	9	200	688	30	892	299	2.5	12	C	Ex.	
4-17	G	ERW	42.7	-	-	-	0	-	-	-	409	43	566	185	11.0	6	P	C. Ex.	
4-18	G	ERW	42.7	650	630	615	11	3	1	80	427	40	570	191	7.0	8	C	C. Ex.	
4-19	J	ERW	42.7	650	600	545	65	14	9	200	552	29	744	248	3.0	0	-	Ex.	
4-20	K	ERW	42.7	750	705	690	65	14	9	200	431	48	611	202	3.0	13	C	Ex.	
4-21	K	ERW	42.7	650	620	615	65	14	9	200	511	33	704	233	3.0	13	C	Ex.	
4-22	K	ERW	42.7	750	710	685	41	7	4	140	425	47	604	206	3.0	12	C	Ex.	
4-23	K	ERW	42.7	950	910	890	22	3	2	110	410	45	570	183	18.0	13	C	C. Ex.	

\* C : cementite, B : bainite, M : martensite, P : Pearlite

Note: Ex. : examples pertaining to the present invention, C. Ex. : comparative examples.

ERW : electric resistance welded pipe.

SP: Seamless Pipe

Sample No. 4-17 did not undergo reducing.

Table 8

No.	Steel No.	Base pipe		Rolling conditions							O.D. of product pipe (mm)	Characteristics of product pipe							Note
		Type	O.D. (mm)	Heating temp. (°C)	Temp. at starting rolling (°C)	Temp. at finishing rolling (°C)	Cumulative diameter reduction (%)	No. of total passes	No. of passes (5% and up per pass)	Final rolling speed (mm/min)		Tensile strength TS (MPa)	Elongation B (%)	High speed tensile strength (MPa)	Energy absorbed at collision and impact (MJ·m <sup>-2</sup> )	Ferrite grain size (μm)	Ratio of area of secondary phase (%)	Kind of secondary phase	
5-1	L	SP-PW	42.7	730	700	640	65	14	9	200	530	43	734	242	2.0	8	C	Ex.	
5-2	L	SP-PW	42.7	670 <sub>1</sub>	640	600	65	14	9	200	640	38	884	301	1.0	7	C	Ex.	
5-3	L	SP-PW	42.7	620	600	560	65	14	9	200	730	32	931	318	2.0	8	C	Ex.	
5-4	L	SP-PW	42.7	—	—	—	0	—	—	—	470	40	640	196	7.0	7	C	C. Ex.	
5-5	L	SP-PW	42.7	850	820	800	65	14	9	200	430	43	592	191	10.0	8	C	C. Ex.	
5-6	L	SP-PW	42.7	670	640	600	11	3	1	80	490	37	666	199	6.0	8	C	C. Ex.	
5-7	M	SP-PW	42.7	700	670	620	41	7	4	140	530	40	724	240	2.5	13	C	Ex.	
5-8	N	SP	110	700	700	690	69	17	15	400	663	42	885	298	1.5	23	C+B	Ex.	
5-9	O	ERW	42.7	720	690	650	65	14	9	200	712	34	931	318	1.5	12	M	Ex.	
5-10	P	ERW	110	700	700	680	77	24	18	400	581	44	802	259	1.5	18	C	Ex.	

\* C : cementite, B : bainite, M : martensite,

Note: Ex. : examples pertaining to the present invention, C. Ex. : comparative examples.

SP-PW : solid-phase pressure-welded pipe, SP : seamless pipe

ERW : electric resistance welded pipe

Sample No. 5-4 did not undergo reducing.

Table 11

Steel No.	Chemical composition (wt%)																		Ac <sub>1</sub> (°C)	Note
	C	Si	Mn	P	S	Al	N	O	Cu	Ni	Cr	Mo	V	Nb	Ti	B	Ca	REM		
V	0.09	0.02	0.73	0.011	0.003	0.032	0.0036	0.0025	—	—	—	—	—	—	—	—	—	—	770	Ex.
W	0.11	0.15	1.28	0.007	0.001	0.028	0.0041	0.0025	0.12	0.16	0.15	—	—	—	—	—	—	—	755	Ex.
X	0.14	0.35	0.91	0.008	0.001	0.025	0.0038	0.0033	—	—	—	—	0.02	0.021	0.007	0.0011	—	—	770	Ex.
Y	0.12	0.25	1.36	0.008	0.001	0.028	0.0030	0.0028	—	—	—	—	—	—	—	—	0.003	—	760	Ex.
Z	0.21	0.20	0.48	0.009	0.001	0.025	0.0038	0.0031	0.12	0.12	0.11	0.05	0.02	0.009	0.009	—	—	0.006	765	Ex.

Table 12

No.		Steel No.	Base pipe		Rolling conditions						O.D. of product pipe (mm)	Characteristics of product pipe								Note	
			Type	O.D. (mm)	Heating temp. (°C)	Temp. at starting rolling (°C)	Temp. at finishing rolling (°C)	Cumulative diameter reduction (%)	No. of total passes	No. of passes (5% and up per pass)		Yield strength (MPa)	Tensile strength TS (MPa)	Elongation (%)	High speed tensile strength (MPa)	Energy absorbed at collision and impact (MJ/m <sup>2</sup> )	Fatigue strength (MPa)	Ferrite grain size (µm)	Ratio of area of secondary phase (%)		Kind of secondary phase
7-1		V	ERW	110	660	650	630	68	14	9	35.0	466	550	47	742	198	220	1.5	14	C	Ex.
7-2		V	ERW	35	—	—	—	—	—	—	35.0	364	448	45	553	124	140	13.0	15	C	C. Ex.
7-3		W	ERW	110	605	600	590	68	14	9	35.0	531	612	40	821	223	250	1.5	18	C	Ex.
7-4		W	ERW	110	880	860	830	68	14	9	35.0	421	517	38	648	143	155	8.0	16	C+B	C. Ex.
7-5		W	ERW	110	660	650	640	18	4	2	90.0	451	522	36	679	151	160	9.0	18	C	C. Ex.
7-6		X	SP	110	660	650	630	77	17	10	25.6	507	596	40	795	196	235	2.0	16	C	Ex.
7-7		Y	SP	110	660	650	630	77	17	10	25.6	523	618	39	806	198	240	2.5	20	C	Ex.
7-8		Z	SP	110	660	650	630	77	17	10	25.6	570	657	37	850	210	255	2.0	23	C	Ex.

Note: Ex. : examples pertaining to the present invention, C. Ex. : comparative examples.

ERW : electric resistance welded pipe, SP : seamless pipe

\* C : cementite, B : bainite, M : martensite,

\*\* 0.2% PS

\*\*\* load stress for 10<sup>6</sup> cycles of endurance.

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more than 0.02% and Ca not more than 0.01%, with the remainder being Fe and unavoidable impurities.

16. A process for producing a steel pipe having high ductility and high strength, said process comprising heating a base steel pipe at a temperature of ( $A_{c1} + 50^{\circ}\text{C}$ ) to  $400^{\circ}\text{C}$  and subsequently performing reducing on it at a rolling temperature of ( $A_{c1} + 50^{\circ}\text{C}$ ) to  $400^{\circ}\text{C}$  such that the cumulative reduction of diameter is 20% or more, said steel pipe stock having a composition of C : 0.06-0.30%, Si : 0.01-1.5%, Mn : 0.01-2.0%, and Al : 0.001-0.10% on a weight basis, with the remainder being Fe and unavoidable impurities.
17. A process for producing a steel pipe as defined in Claim 16, wherein said composition further contains one or more selected from Cu not more than 1.0 %, Ni not more than 2.0%, Cr not more than 2.0%, and Mo not more than 1.0% on a weight basis.
18. A process for producing a steel pipe as defined in Claim 16 or 17, wherein said composition further contains one or more selected from Nb not more than 0.1 %, V not more than 0.3%, Ti not more than 0.2%, and B not more than 0.004% on a weight basis.
19. A process for producing a steel pipe as defined in any of Claims 16 to 18, wherein said composition further contains one or more selected from REM not more than 0.02 % and Ca not more than 0.01% on a weight basis.
20. A process for producing a steel pipe as defined in any of Claims 14 to 19, wherein the heating temperature is  $750-400^{\circ}\text{C}$  and the rolling temperature is  $750-400^{\circ}\text{C}$ .
21. A process for producing a steel pipe as defined in any of Claims 14 to 20, wherein reducing is performed such that at least one of rolling passes reduces the diameter by 6% or more per pass.
22. A process for producing a steel pipe as defined in any of Claims 14 to 21, wherein reducing is performed such that the cumulative reduction of diameter is 60% or more.
23. A process for producing a steel pipe as defined in any of Claims 14 to 22, wherein reducing is performed in the presence of a lubricant.

FIG. 3

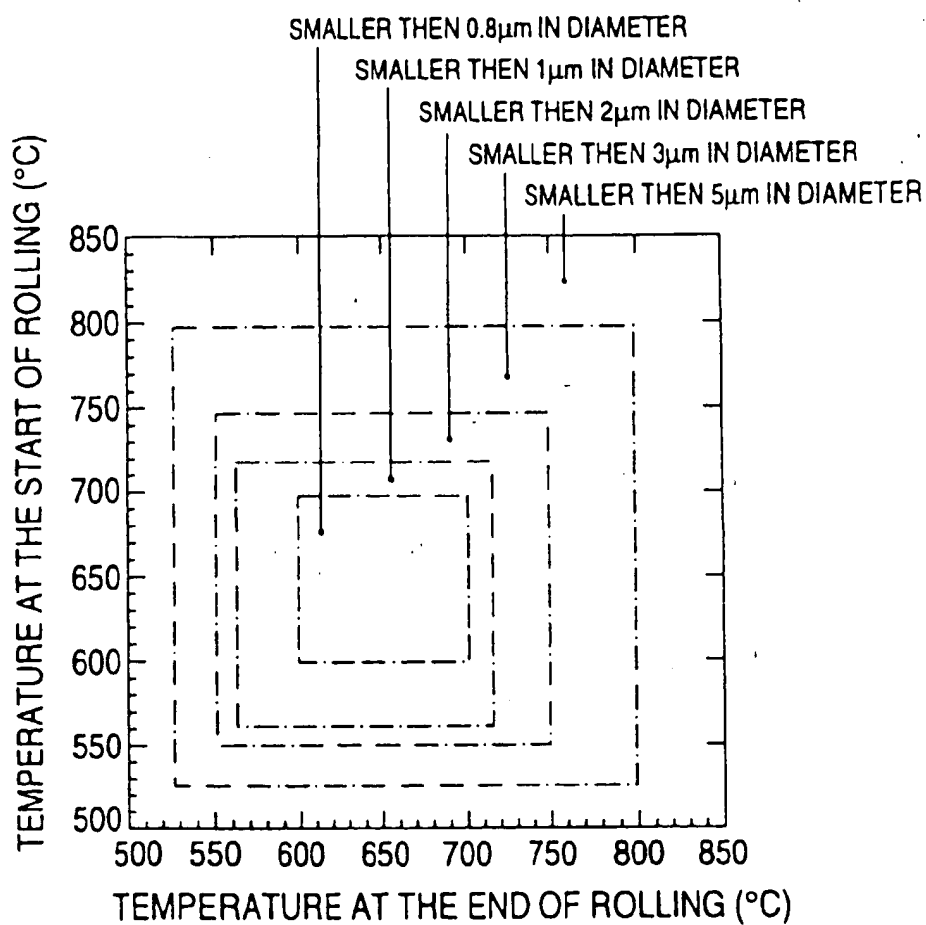
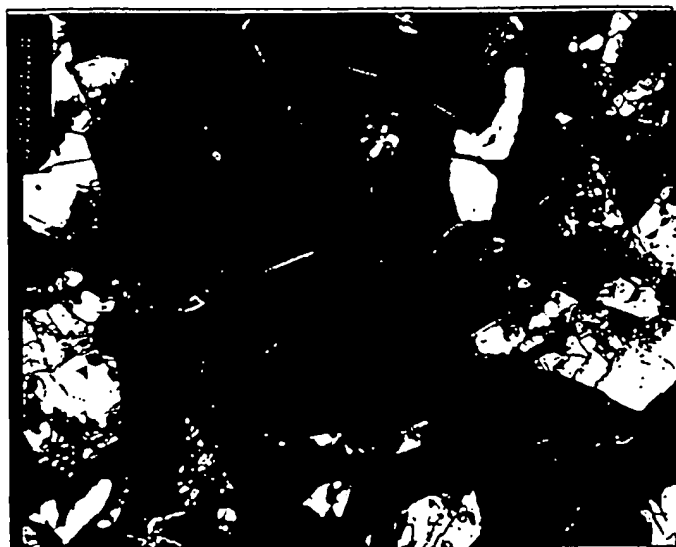


FIG. 4



0.5 $\mu$ m

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP98/01924

## A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl<sup>1</sup> C22C38/00, 38/14, 38/44, 38/54, C21D8/00, 8/10

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int.Cl<sup>1</sup> C22C38/00-38/60, C21D8/00-8/10

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho 1926-1996 Toroku Jitsuyo Shinan Koho 1994-1998  
 Kokai Jitsuyo Shinan Koho 1971-1998 Jitsuyo Shinan Toroku Koho 1996-1998

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	JP, 9-279233, A (Nippon Steel Corp.), October 28, 1997 (28. 10. 97) (Family: none)	1-23
X	JP, 8-60239, A (Nippon Steel Corp.), March 5, 1996 (05. 03. 96) (Family: none)	1, 2, 6
X	JP, 8-3679, A (Nippon Steel Corp.), January 9, 1996 (09. 01. 96) (Family: none)	1, 2
X	JP, 2-301540, A (Sumitomo Metal Industries, Ltd.), December 13, 1990 (13. 12. 90), Claims ; Tables 1 to 4 & US, 5080727, A & EP, 372465, A1	1-4, 12
A	JP, 9-49050, A (Kobe Steel, Ltd.), February 18, 1997 (18. 02. 97) (Family: none)	1-23
A	JP, 3-267316, A (Sumitomo Metal Industries, Ltd.), November 28, 1991 (28. 11. 91) (Family: none)	1-23

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

\* Special categories of cited documents:

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